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A COMPARISON OF NEUTRON-ACTIVATION AND CHEMICAL TECHNIQUES FOR --ETC(U)

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A COMPARISON OF NEUTRON-ACTIVATION AND  
CHEMICAL TECHNIQUES FOR DETERMINING THE  
FLUORINE CONTENT OF FLUOROCHEMICAL-TREATED  
FABRICS

by

F.A. Johnson and J.M. McAndless



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A COMPARISON OF NEUTRON-ACTIVATION AND CHEMICAL TECHNIQUES  
FOR DETERMINING THE FLUORINE CONTENT OF FLUORO-CHEMICAL-TREATED FABRICS

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ABSTRACT

The fluorine content of fabric samples treated with three different fluorochemical finishes has been determined by means of both chemical and neutron-activation techniques. The results obtained using the two analytical techniques are compared, and the advantages of neutron activation are discussed.

RÉSUMÉ

On a déterminé la teneur en fluor des tissus traités avec différents finis fluoro-chimiques en procédant à la fois par analyse chimique et par activation neutronique. On compare les résultats obtenus avec ces méthodes analytiques, et on discute des avantages de l'activation neutronique.

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## INTRODUCTION

DREO has been actively engaged in studying and developing oil- and water-repellent textile finishes suitable for use on specialized clothing employed by the Canadian Forces. Certain fluorine-containing polymers are especially effective in providing the desired protective properties and are also useful in augmenting the overall effectiveness of less expensive hydrocarbon-based water repellents. Such "fluorochemical" finishes are usually applied by wetting the fabric with a solution of the fluorine-containing polymer in an organic solvent, or with a dispersion of the polymer in water, and then drying and curing under controlled conditions. In this way the constituent fibres of the fabric become coated with a coherent film of the fluoropolymer.

Methods of measuring the fluorine content of such treated fabrics are of interest in order to confirm that manufacturing specifications have been followed, and to determine the durability of the finish during wear or in the laundering process. In addition, such measurements have made it possible to relate the loss of repellent properties after laundering to a probable disorientation of the fluoropolymer substituents at the surface of the finish rather than to a significant loss of the finish material itself (1).

Chemical methods of fluorine analysis, as applied to treated fabrics, are generally hampered by the extreme resistance of most fluoropolymers to complete decomposition (2). In addition, such methods of analysis are destructive of the fabric and so do not permit continuing tests of the same sample to be carried out. Neutron activation analysis, on the other hand, is a non-destructive technique which can be adapted to analysis of the fluorine content of a treated fabric sample, and procedures employing this method of analysis have recently been developed at DREO.

This report compares the results of fluorine analyses of a selection of treated fabrics as obtained by means of both chemical and neutron activation techniques. The fabric samples employed were nylon/cotton (50/50) twist fabrics weighing  $270 \text{ g/m}^2$  ( $5 \text{ oz/yd}^2$ ) which had been treated with various commercially available fluorochemical finishes. The amount of any fluorochemical applied to these fabrics was such that the fluorine content of the resulting finish was less than 1% by weight of the fabric.

MATERIALS AND METHODSFinishes

The fluorochemical finishes selected for study were all obtained from commercial sources and used as received. These finishes are listed in Table I.

TABLE IFluorochemical Finishes Studied<sup>a</sup>

Name	Chemical Description	Source of Supply
FC-232	Water-based fluoropolymer	3M Company
Zepel B	Water-based fluoropolymer	Dupont
Tinotop T-10	Solvent-based two-component finish containing a fluoro-polymer and methacrylate adjunct.	Ciba-Geigy

<sup>a</sup> Use of a commercial product does not imply recommendation or approval of that product by the Department of National Defence to the exclusion of other products.

Fabric Treatment

A 50/50 nylon/cotton twist fabric, dyed but otherwise unfinished, weighing 270 g/m<sup>2</sup> (5 oz/yd<sup>2</sup>), was used as the fabric substrate for all experiments.

Appropriate treatment baths containing the finishes listed in Table I were made up in accordance with the manufacturer's technical brochure associated with each finish. The FC-232 and Zepel B treatment baths contained 3% solids by weight, while the Tinotop T-10 bath, which employed perchloroethylene as solvent, contained 1% solids by weight.



Using a two-dip/two-nip procedure, a number of fabric samples were padded on an Atlas padding machine, Type LW-1, to a wet pick-up of 70-100% with each of the finishes. Following padding, the fabrics were dried at room temperature and then cured at 145°C for eight minutes.

The same fabric substrate had been treated previously in commercial mills with each of the finishes listed in Table I under similar conditions to those used in the laboratory. These commercial samples were included in the fluorine analysis experiments for comparison purposes.

### ANALYTICAL TECHNIQUES

#### Chemical Method

The chemical method employed at DREO for determining the fluorine content of treated fabrics is similar to the standard AATCC procedure (3) for fluorine determination.

One-inch-square sample pieces are cut from the fabric, weighed, and then completely burned in air in a 500-ml Schöniger Combustion Flask containing 10-20 ml of water. Four separate combustions of single one-inch fabric squares are carried out and the aqueous solutions which contain the fluorides resulting from each combustion are combined. The combined solution is then titrated with 0.02N thorium nitrate using sodium alizarin sulfonate as an indicator. The entire combustion/titration procedure is repeated a total of three times to give three replicate determinations.

The weight of titrated fluorine is determined by reference to a calibration graph. This graph is produced by titrating known amounts of lithium fluoride with 0.02N thorium nitrate using sodium alizarin sulfonate as the indicator.

The fluorine content of a given fabric sample is calculated by dividing the total weight of titrated fluorine resulting from the 12 combustions by the combined weight of the 12 one-inch-square samples and expressing the result as a percentage.

The error associated with this method is normally 1 to 5%, with the measured fluorine content usually lower than that known to be present. The major contribution to this error results from incomplete combustion of the very-high-molecular-weight fluorinated polymers present in the finish, as indicated previously.



In a variation of this procedure, a fluoride-sensitive electrode coupled to a pH meter with an expanded scale can be used to replace the titration step. Once the meter has been calibrated with solutions of known fluoride concentration, the concentration of fluoride in the combustion solution may be read directly.

#### Neutron Activation Method

As will be described in more detail in a forthcoming report (4), consideration of the various fast-neutron-induced reactions by which fluorine can be activated led to the choice of the  $^{19}\text{F}(\text{n},2\text{n})^{18}\text{F}$  reaction as being the most suitable for the determination of fluorine in fabric samples. In particular, this reaction is least subject to interference by reactions produced in the constituent atoms of the fabric or by other activities produced in fluorine. The same reaction has also been previously considered for the determination of the fluorine content of Quarpel-treated fabrics (5), but in this case an inconsistency in some of the data was noted.

The isotope  $^{18}\text{F}$  is a positron emitter with a half-life of about 110 minutes, and measurement of the amount of  $^{18}\text{F}$  formed depends on detection of the 0.511-MeV  $\gamma$ -rays which are produced as the emitted positrons come to rest and annihilate with ordinary electrons in the material surrounding the activated sample. Two such  $\gamma$ -rays are emitted in each annihilation process and both of these could be detected in a coincidence arrangement involving two scintillation detectors, or else only one of the two quanta could be detected with a single detector. In the present instance, since only a single activity was to be measured, and because the spectrum was not complex, a single such well-shielded detector was found to be adequate. The main interference was expected to be due to the 10-minute positron emitter  $^{13}\text{N}$  produced by the  $^{14}\text{N}(\text{n},2\text{n})^{13}\text{N}$  reaction in the nylon component of the fabric. Such interference with the determination of  $^{18}\text{F}$  can be effectively avoided by delaying the start of counting until 2-2½ hours after the end of the neutron irradiation, at which time the contribution of  $^{13}\text{N}$  to the measured activity will have become insignificant.

Activation of the coated fabrics was carried out using 14.9-MeV neutrons produced in the  $^3\text{H}(\text{d},\text{n})^4\text{He}$  reaction by the DREO 150-keV Cockcroft-Walton neutron generator. A total neutron output of about  $10^{10}$  n/sec was generally attained.

The fabric samples to be analyzed by activation were cut into circular discs 28.6 mm (1-1/8 inch) in diameter which weighed about 100-110 mg each and were 0.23 to 0.36 mm (9 to 14 mils) thick. Prior to irradiation, the total number of fabric discs which were to be activated was collectively sealed in polyethylene-coated Mylar film, 0.038 mm (1.5 mils) thick (0.75 mil polyethylene + 0.75 mil Mylar), in order to retain recoil  $^{18}\text{F}$  nuclei which otherwise could escape from the samples during irradiation. The sealed fabric samples were sandwiched between two similarly sealed discs of 0.051 mm (2 mil) Teflon film. The Teflon discs served as reference standards containing a known amount of fluorine (76%) and were activated at the same time as the fabric in essentially the same neutron flux. This sandwich of the materials to be activated was held rigidly between two discs of polystyrene 2.4-3.2 mm (3/32-1/8 inch) thick which were bolted together, and the whole unit was placed directly on the water-cooled target cap of the neutron generator. Details of the polystyrene holder and the components of the sandwich are shown in Figure 1.

After irradiation, the sealed fabric and Teflon samples were separated and the activity of each was determined by means of a 7.6 cm x 7.6 cm (3" x 3") integrally mounted NaI(Tl) scintillation detector and a single-channel analyzer set to cover the whole of the 0.511-MeV photopeak. The sealed samples were each counted in turn for periods of 10-15 minutes and the decay of each was monitored for a length of time sufficient to establish the presence of the characteristic 110-minute half-life of  $^{18}\text{F}$ . The fluorine content of a given fabric sample was then determined in the usual manner by comparing its activity with that of the average specific activity of the two Teflon standards.

## RESULTS AND DISCUSSION

Table II gives the fluorine content of the fabric substrate treated with different fluorochemical finishes as determined by chemical and neutron-activation analyses. In each case, the percentage fluorine by weight represents the total fluorine contributed by the fluorochemical finish. This value is substantially less than the total weight of finish added on to the fabric (approximately 3% weight add-on in the case of the water-based finishes and 1% weight add-on for the solvent-based finish), since these finishes contain non-fluorinated moieties or adjuncts which do not contribute to the fluorine content of the finish.

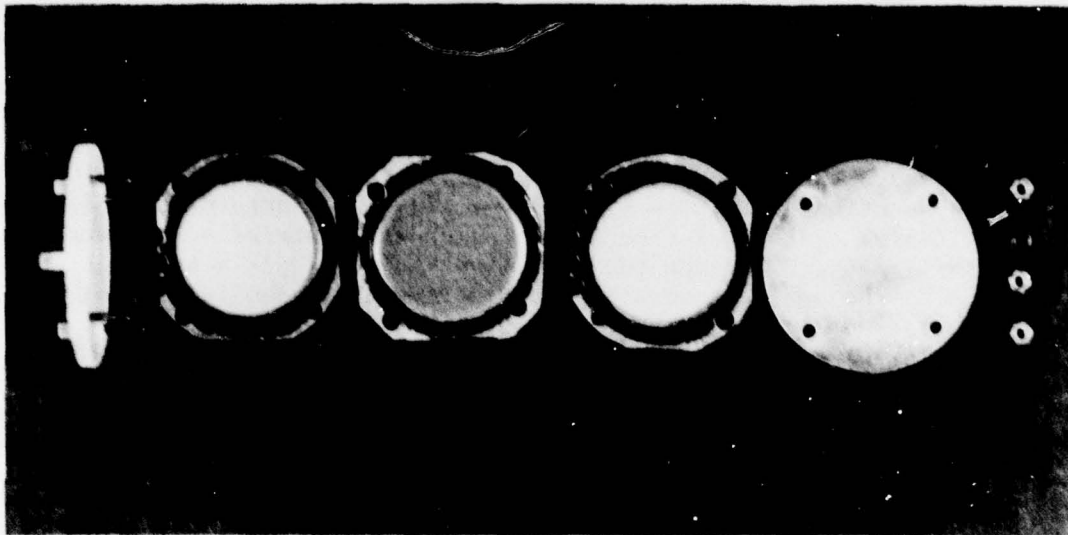


Figure 1:

Components of the polystyrene sample holder and the elements of the sandwich. From left to right: base of holder which sits on the target cap of the neutron generator; front Teflon standard sealed in Mylar; fabric sample sealed in Mylar; rear Teflon standard sealed in Mylar; rear plate of holder; nuts to fit bolts protruding from base of holder in order to hold the sandwich together. The indentations in the Mylar of the Teflon standards are for purposes of identification.



In general, the two sets of analytical results given in Table II indicate that those fabrics which had been finished in the laboratory show a consistently higher fluorine content than do those which had been treated commercially. This difference can probably be accounted for by the fact that the laboratory-treated fabrics were padded at a much slower speed than that employed during the commercial process, thereby enabling the fabrics to be more thoroughly wetted by the finishing solution and yielding a higher net pick-up of the fluoropolymer.

The results of the fluorine determinations using the neutron activation technique, as presented in Table II, were all based on irradiation of three sealed fabric discs. The quoted result in each case represents the weighted mean and the weighted standard deviation (based on counting statistics alone) of at least ten determinations of the fluorine content of each sample which were carried out as the  $^{18}\text{F}$  activity decayed through two half lives or more. In a number of cases (A, B and C) the analyses were repeated using only a single disc of the fabric concerned; in these cases, the results obtained were in accord with those given in Table II. It would be expected, however, that a fluorine analysis based on the measured activity of a stack of three discs would give a consistently lower value than that derived using a single disc, both because of the progressively greater distance of each disc from the detector surface and also because of  $\gamma$ -ray absorption in the underlying discs. Preliminary measurements suggest that this effect amounts to less than 2% for the case of three discs and hence would tend to be masked by the comparable uncertainty attributable to counting statistics.

The fluorine values determined by the two different analytical techniques are generally in agreement with one another within the limits of the quoted uncertainties, although in some cases there is an evident tendency for the chemical results to be somewhat lower than those obtained by neutron activation. While such a tendency was not unexpected, for reasons already discussed, it should be pointed out that the samples which were analyzed by the two techniques were not identical, being taken from different areas of the fabric roll. Thus, the uniformity of the fluorochemical finish over the area of the fabric would be an important factor affecting the agreement to be expected between the results obtained by the two analytical methods. The greater experimental accuracy (generally 1-2%) attainable with the activation as compared with the chemical technique suggests that neutron activation could provide a useful means of investigating the detailed uniformity of the fluorochemical finish over an area of a treated fabric.



TABLE I

Fluorine Content of a Nylon/Cotton Fabric Treated  
With Different Fluorochemical Finishes

Fabric Identification	Finish	Source of Treatment	% Fluorine by Weight of Fabric			
			Schöniger Combustion		Neutron Activation	
A	FC-232	Laboratory	0.83 ± 0.03		0.830 ± 0.006	
B	FC-232	Commercial	0.71	0.03	0.693	0.006
C	Zepel B	Laboratory	0.46	0.01	0.502	0.005
D	Zepel B	Commercial	0.40	0.01	0.420	0.005
E	Zepel B	Commercial	0.43	0.01	0.434	0.008
F	Tinotop T-10	Laboratory	0.25	0.01	-	
G	Tinotop T-10	Commercial	0.24	0.01	0.242	0.004
H	None	-	0.02 <sup>b</sup>		0.0 <sup>c</sup>	

<sup>b</sup> Within the experimental error associated with the chemical analysis technique.

<sup>c</sup> No distinct evidence of <sup>18</sup>F half-life.

Thus, it is apparent that neutron activation analysis has a number of distinct advantages over the chemical technique. Apart from the higher potential accuracy in the method, the technique avoids the inherent uncertainty involved in obtaining complete combustion of the sample in order to convert the fluorine to an inorganic form suitable for chemical manipulations. In addition, a smaller sample of the fabric can be employed than is required for analysis by chemical means. Being a non-destructive technique, neutron activation will permit analyzed samples to be replaced in the fabric substrate (e.g. by stitching) in order that further tests, such as laundering or wearing, can be carried out, after which the same sample can be removed and analyzed again.

The reliability of the neutron activation technique, as developed at DREO, depends largely on effective retention of those active recoil  $^{18}\text{F}$  nuclei which have sufficient kinetic energy to escape from either the fabric samples or the Teflon standards. It is probable that the loss or migration of these active nuclei was responsible for the inconsistency in some of the data which was noted in previous work (5) which employed the same nuclear reaction and a similar technique.

In neutron activation analysis the fall-off in the neutron flux away from the target is of concern when a sandwich arrangement of sample and standards is employed, since different specific activities will be induced in the successive components of the sandwich. In the present instance, the specific activities of the two Teflon standards could differ by up to 11% (for the case of three fabric discs interposed) as a result of the flux gradient through the sandwich. The fact that the analytical results appeared to be independent of the number of fabric discs on which the determinations were based implied that the flux gradient was essentially linear across the thickness of the sandwich. Thus, in this case, the average specific activity of the two Teflon standards provided a meaningful measure of the neutron flux at the position of the fabric sample. This technique, however, has its limitations if the sandwich is too thick because, as well as the expected inverse-square variation at larger distances, the neutron flux falls off so rapidly near the target that there is potential for an inaccurate estimate of the average flux. More uniform irradiation of the components of the sandwich could be attained by rotation of the sandwich about an axis perpendicular to the axis of the deuteron beam so that, on average, the sandwich would be irradiated equally from both sides, and the flux fall-off would be effectively compensated for.

### CONCLUSIONS

1. Neutron activation and Schöniger combustion techniques for determining the amount of fluorine present in fluorochemical-treated fabrics give results which are in general agreement. The activation technique, as well as requiring a smaller sample for analysis, can provide greater experimental accuracy (~1-2%) as compared to the chemical technique (~1-5%), and is free from inherent uncertainties peculiar to the chemical method.
2. Neutron activation, unlike the chemical technique, is non-destructive and so permits continuing tests to be carried out on the same fabric sample.
3. The potential accuracy of the neutron activation technique would permit an investigation of the detailed uniformity of a fluorochemical finish over an area of a coated fabric.

### ACKNOWLEDGEMENTS

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